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Preconcentration of Sunset Yellow Dye using β-Cyclodextrin Epichlorohydrin Polymer as the Solid Phase Extractant

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Abstract: ***A solid phase extraction method has been developed for the preconcentration of Sunset yellow dye at the trace level using β-Cyclodextrin polymer. After preconcentration the dye solute was determined UV-VIS spectrophotometry. Various parameters, such as effect of pH, sample volume, shaking time, amount of adsorbent, agitation speed for the % uptake of Sunset yellow has been optimized. This method has been applied for the determination of Sunset yellow in different food samples.***
Keywords: β-Cyclodextrin, β-Cyclodextrin epichlorohydrin polymer, Sunset yellow dye, preconcentration, Spectrophotometry.

I. INTRODUCTION

Industrialization of food systems, including food processing, has increased the use of food additives such as food dyes, preservatives and sweeteners. Many industries such as pharmaceutical, plastic, paper, textile and cosmetics etc. use dyes in order to color their products. Sunset Yellow, a synthetic dye, which is used in fermented foods which must be heat treated. It may be found in orange sodas, marzipan, Swiss rolls, apricot jam, citrus marmalade, lemon curd, sweets, beverage mix and packet soups, custard powders, packaged lemon gelatin deserts, pharmaceutical pills and other products with artificial yellow, orange or red colors. Sunset Yellow is used commercially as alimentary additives, in pharmaceuticals and cosmetics, with the advantages that they can be easily mixed to achieve ideal colors and because of their low price compared with the natural dyes. Sunset yellow may be responsible for causing an allergic intolerance[1], resulting in various symptoms, including diarrhea, vomiting, swelling of skin and migraines[2-3]. Sunset Yellow may have immunomodulatory effects[4]. The analytical techniques frequently employed for the determination of the colors include spectrophotometry [5-8] , column solid-phase extraction[9], derivative spectrophotometry[10], HPLC[11], kinetic determination[12], second derivative spectrophotometry[13], adsorptive sripping voltammetric[14]. In all instances, most of these methods require a highly qualified operator and high cost instrumentation. Thus, a highly sensitive and low cost method is still needed for the development in the field of analytical chemistry. So, Sunset yellow dye has been determined by spectrophptometric methods after preconcentation using β-CDP in food samples.

Supramoleculer complexes with β- cyclodextrin has been a very active research field in the past few years[15-16]. β- cyclodextrin (β-CD) is a very stable oligosaccharide that is composed of seven glucose units linked with each other by α-(1,4)-glycosidic linkage. It can form supramoleculer complexes with several organic compounds by incorporating them into their hydrophobic cavities. Two or more β-cyclodextrin covalently linked with each other are known as polymers. These β- cyclodextrin polymer have been used for the preconcentration of various analytes[17-20]. In the present work, β-cyclodextrin epicholorohydrin polymer (β-CDP) has been used as a solid support for the preconcentration of Sunset yellow dye.

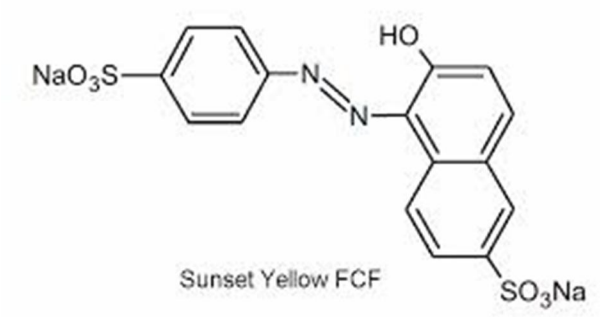


Fig. 1: Structure of Disodium 6- hydroxy – 5- [(4-sulfophenyl)azo] – 2 –naphthalenesulfonate (Sunset Yellow Dye)

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B. Experimental

A Shimadzu UV-1800 spectrophotometer (Shimadzu Ltd., Japan) equipped with the matched 10 mm quartz cells was used to measure absorbance. Digital century pH-meter Cp - 901 with a combined glass electrode was used to carry out pH measurements. A thermostatic shaking water bath (Perfit India Ltd.) was used to carry out all the inclusive procedures.

C. Reagents

All chemicals used were of AnalR grade unless otherwise stated. Double distilled water wasused throughout the experiment. Brilliant green dye solution was prepared by dissolving 0.248 g in 100 mL of double distilled water to give 0.01 M standard stock solution and further diluted as and when required. β-CDP was synthesized by method given in Literature (1[a]). A brief procedure is given here : 40g of β-CD, 10 g of soluble starch and 100 mL of 20% sodium hydroxide were added into a beaker. The mixture was vigorously stirred at 50-60ºC until the reactants dissolved. Total 60 mL of epichlorohydrin was added drop wise into the solution, and β-CDP was formed in 30 min. Filtered with pressure through Buchner funnel and then washed with distilled water 5-6 times, the polymer was dried at 100ºC and then stored at room temperature in the desiccators for further use.

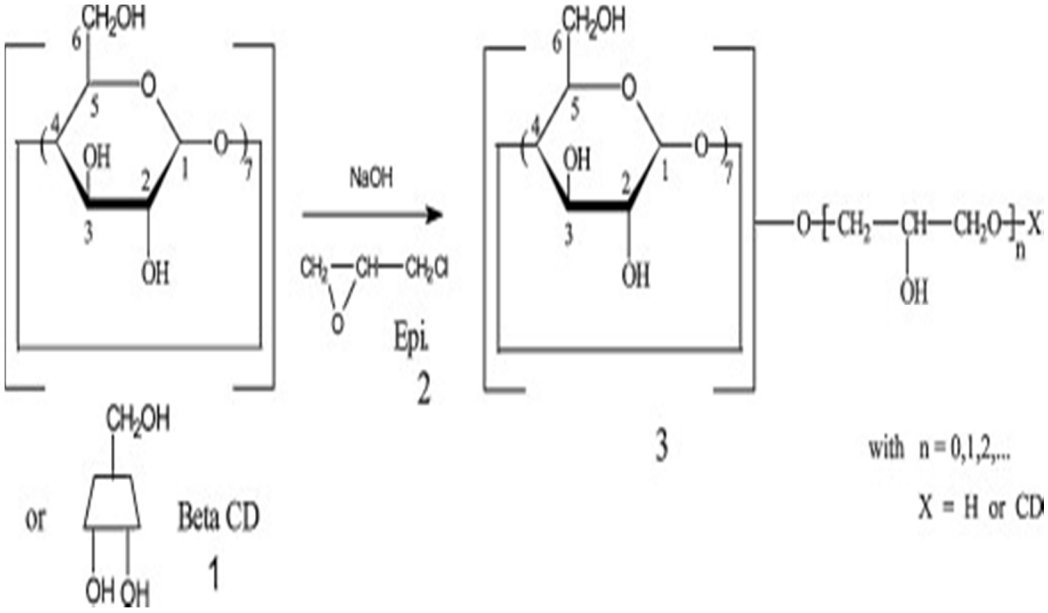


Fig.2: Schematic representation of the synthesis of β-cyclodextrin polymer

Buffer solution in the pH range of 2.0-3.5 were made by mixing equimolar solutions of hydrochloric acid/Sodium Acetate and buffer solutions in the pH range of 4.0-6.5 were made by mixing equimolar solutions of sodium acetate and acetic acid solutions in the different proportions While those in the pH range of 7.0-11.0 were made by mixing equimolar solutions of ammonia and ammonium chloride. The glass wares were washed with chromic acid and soaked in 5% nitric acid and then cleaned with double distilled water before use and dried in an electric oven.

D. Procedure

200 mg of β-CDP and 2.5 mL of buffer solution (pH 3.0) were added to a 100 ml stoppered conical flask at room temperature. The mixture was allowed to stand for 15 min. so that β-CDP should swell sufficiently and an appropriate amount of dye was added and made up to 75 ml with double distilled water. The mixture was shaken in the thermostatic shaking water bath for 90 min., at the rate of 140 r.p.m., 5.0 ml of supernatant solution was transferred into a test tube and the absorbance was measured spectrophotometrically

E. Optimization of various parameters

1) *Effect of Ph:* The formation of inclusion complex of the dye in the polymer depends on the pH of the sample solution which was studied in the range of (1.0-7.0.) using different buffer solutions (Fig. 3), % uptake (≥ 95) was obtained at pH 3.0. Therefore, the working pH was chosen as 3.0 for the subsequent studies.

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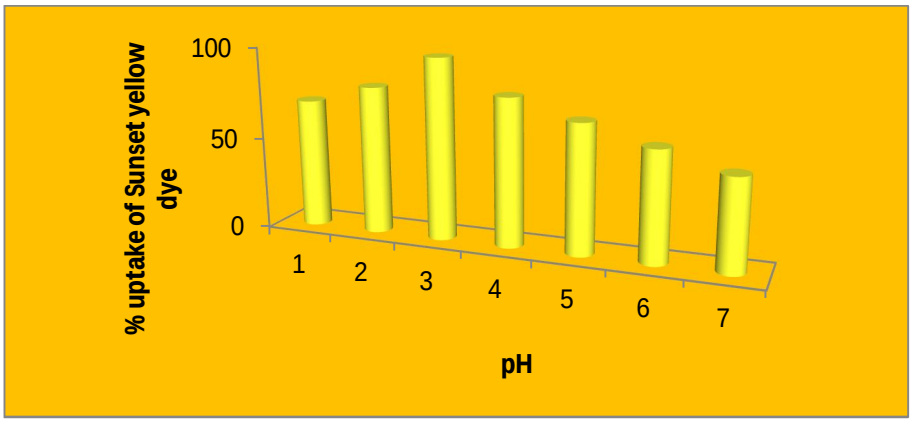


Fig.3: Effect of pH on the % uptake of the Sunset yellow dye by the β-CD polymer.

F. Effect of shaking Time

Shaking time is an important factor in determining the possibility of application of the β-CD polymer for the selective uptake of sunset yellow dye. Different shaking time (ranging from 15 to 105 min.) were studied for the % uptake of sunset yellow dye by β-CD polymer. The results of % uptake of Sunset yellow dye vs. the shaking time show that the % uptake of (≥95%) was attained within 90 min.(Fig.4). Therefore, the shaking time of 90 min. was selected for further studies.

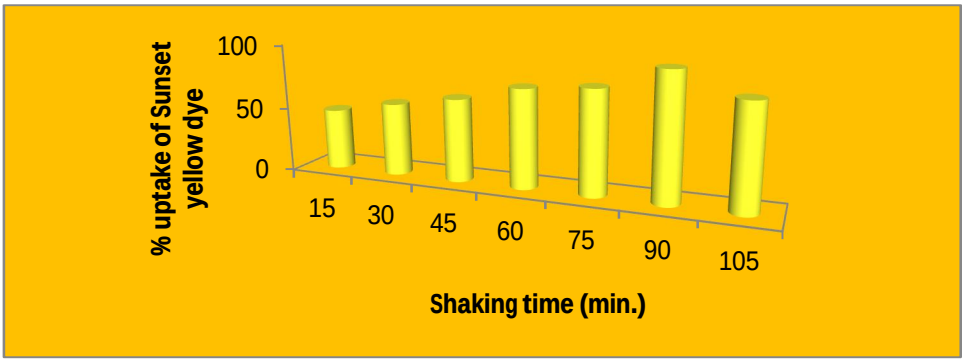


Fig.4:Effect of shaking time on the % uptake of the Sunset yellow dye by the β-CD polymer

G. *Effect of sample volume*

Enriching low concentration of dye from large volume of sample the effect of sample volume is an important factor in determining the possibility of application of polymer for the % of uptake of sunset yellow dye. For this purpose 15, 30, 45, 60, 75 and 90 ml of sample volumes containing a fixed amount of dye were taken and uptake of sunset yellow dye was studied (Fig. 5). The maximum % uptake (≥95%) of sunset yellow dye was at sample volume of 75ml. Therefore, 75ml of sample volume was used for the further studies.

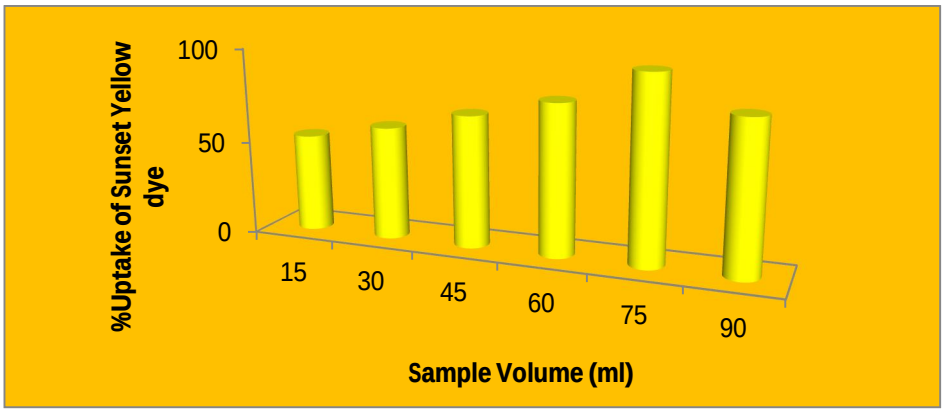


Fig.5: Effect of sample volume on the % uptake of the Sunset yellow dye by the β-CD polymer

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H. Effect of Agitation Speed

Shaking speed is an important factor in determining the possibility of application of polymer for the quantitative % uptake of sunset yellow dye. The driving force i.e Shaking speed could help in mass transfer and facilitate the concentration gradient between the sample solution and the polymer. Different speeds (ranging from 40 to 140 r.p.m.) were studied for the % uptake of Sunset yellow dye by polymer. The results of % uptake of sunset yellow vs. agitation speed (Fig.6) shows that the % uptake reach maximum (≥ 95%) at 140 r.p.m. Therefore, the shaking speed of 140 r.p.m. was selected for further studies.

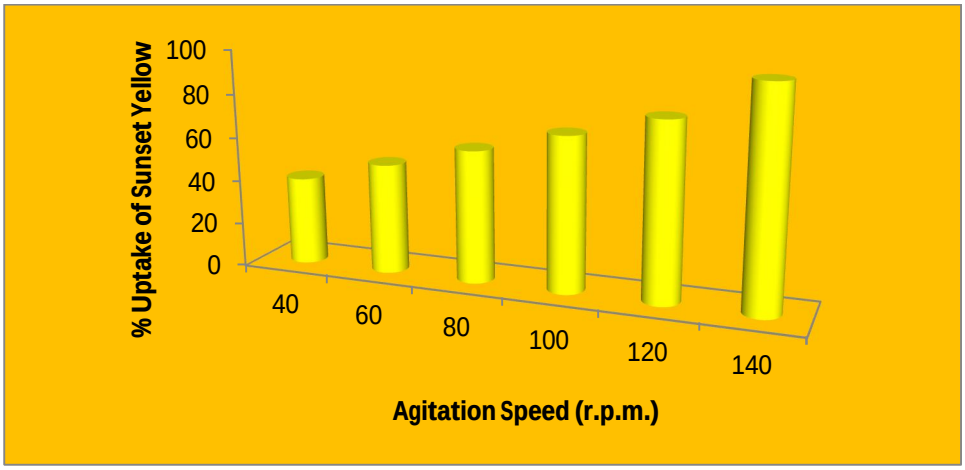


Fig.6: Effect of agitation speed on the % uptake of the Sunset yellow dye by the β-CD polymer.

I. Effect of amount of polymer

The amount of the β-CD polymer is another important parameter that affects %uptake of dye. A quantitative removal (≥ 95%) cannot be achieved when the β-CD polymer is less than the optimum amount. In order to optimize the smallest amount of polymer, 100mg, 200mg, 300mg, and 400mg of the polymer were added to the solution containing known amount of dye. The quantitative recoveries were obtained at 200 mg of β-CDP shown in (Fig.7). Therefore, 200 mg of the β-CDP has been used for further studies.

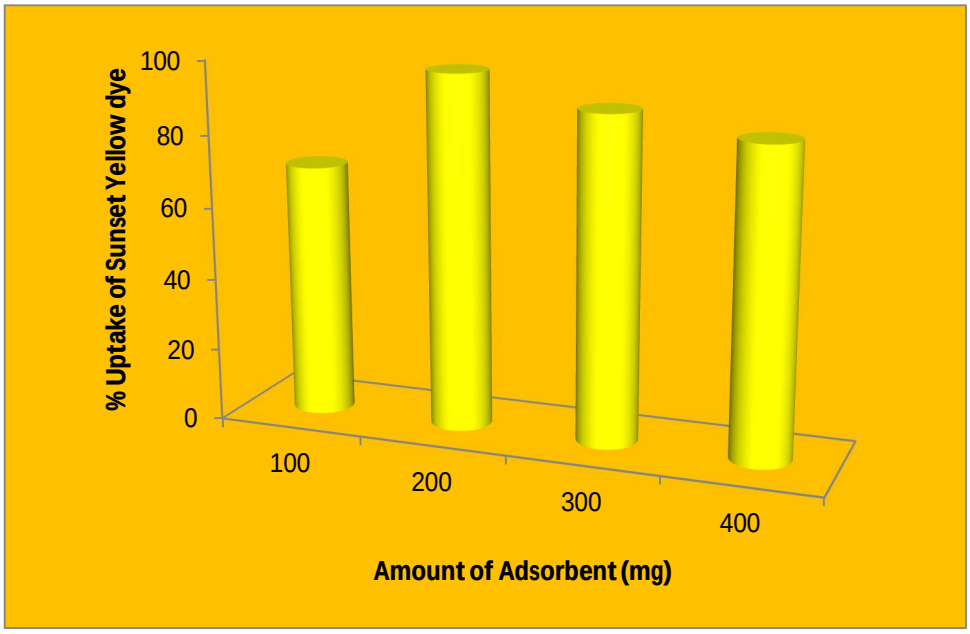


Fig.7: Effect of amount of adsorbent on the % uptake of the Sunset yellow dye by the β-CD polymer

J. Applications

1) Determination of samples: The proposed method has been applied for the determination of Sunset yellow dye in Santra goli, Mrinida and dry syrup (ZIFI 100). The results are given in table.

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Table 1. Result of determination of Sunset yellow in food samples

Food Samples	Added, µg/ml	Found, µg/ml	Recovery, %
[a]Santra Goli	0	0.010	-
	0.304	0.302	96.17
	0.603	0.590	96.24
	1.206	1.208	99.34
[b]Mirinda	0	0.022	-
	0.304	0.301	95.85
	0.603	0.595	95.20
	1.206	1.201	97.80
[a]Dry Syrup (ZIFI 100)	0	0.011	-
	0.228	0.232	97.07
	0.454	0.458	98.49
	0.808	0.810	98.90

[a]Santra goli, [b]Mirinda, [c]Dry Syrup (ZIFI 100) - locally available in market

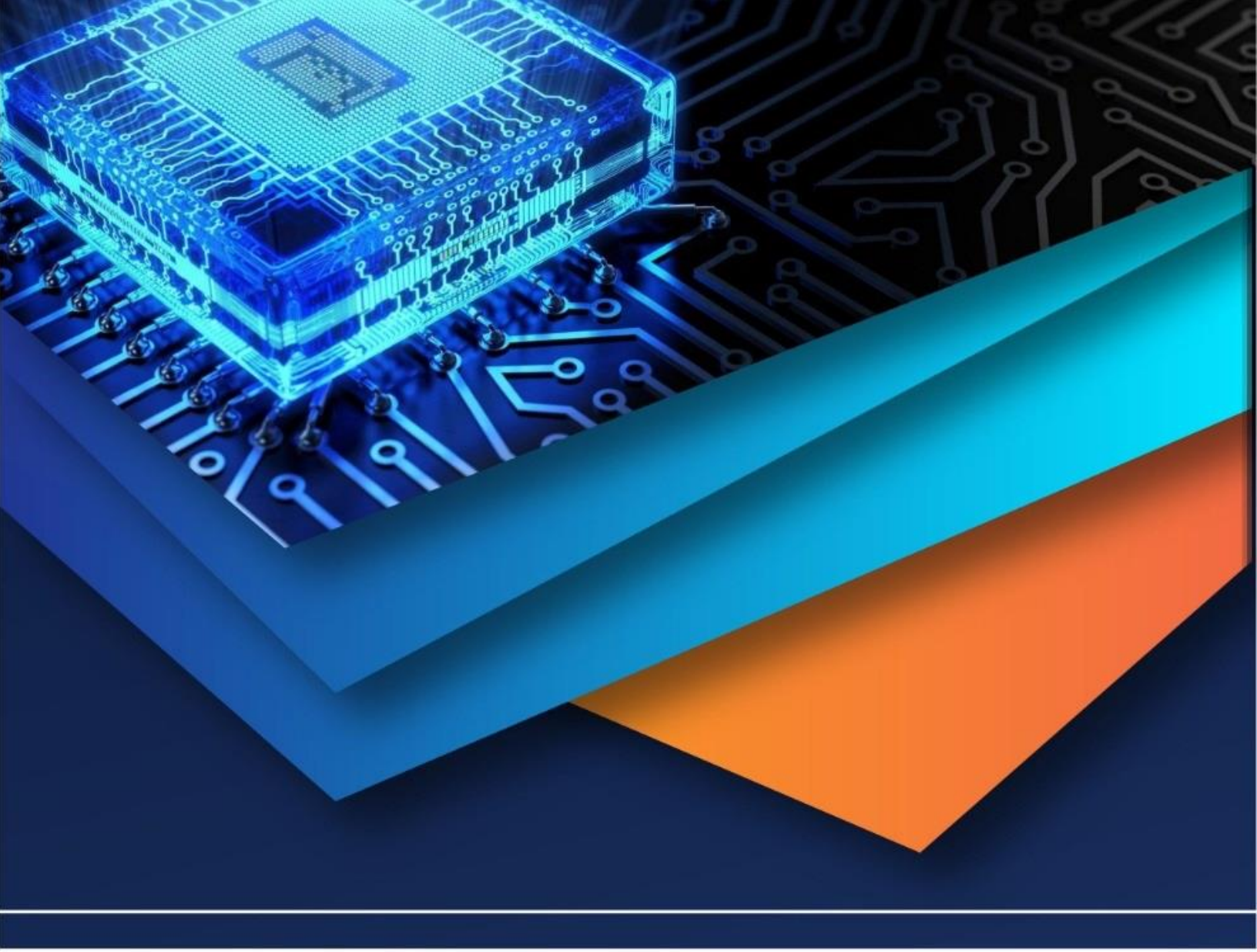
II. CONCLUSION

The proposed preconcentration method consist of a simple and low procedure which permits the quantitative recovery of Sunset yellow dye from food samples. The synthesis of the polymer is easy and the method has a good accuracy, sensitivity and repeatability. The polymer has been used in all the experiments performed for the study. It has a unique stability and reusability. This method is convenient for the determination of Sunset yellow dye.

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